

FW: PCB testing.
WALL Dave to: Bruce Long

03/18/2010 09:50 AM

FYI

----Original Message----

From: Catherine Kopylec [mailto:ckopylec@dexsil.com]

Sent: Wednesday, March 17, 2010 11:29 PM

To: WALL Dave

Subject: Re: PCB testing.

Importance: High

** High Priority **

Hi Dave,

Unfortunately, you can't use any of our products to accurately determine the presence of PCBs in used oils. The Clor-N-Oil kits are only an appropriate test for transformer oil - clean mineral oil - and cannot determine the presence of PCBs directly, rather they measure the amount of chlorine on the PCB molecule. When there is approx. 21 ppm chlorine in the sample, the kit gives a positive result. A positive result in mineral oil is "presumed" to be due to PCBs because there isn't normally any other chlorine to cause interference. But follow up GC lab analysis would be needed to confirm that the positive was, in fact, due to PCBs. Used oils on the other hand always contain some background chlorine - even virgin oil can contain up to 200 ppm. Therefore, you would always expect to get a positive result with the Clor-N-Oil kits - however, in most cases used oil can contain a lot of water that can cause a false negative result. (Water over 5% will cause false negative results with the Clor-N-Oil kits. Clor-D-Tect can handle up to 20 % water.)

The Clor-D-Tect kits would technically pick up the chlorine from PCBs, but because of the possibility of background chlorine from additive packages, as well as the possibility of chlorinated solvents, there is no way to determine that a positive result with a Clor-D-Tect kits is due to PCBs. Further, for a Clor-D-Tect 1000 kit to show a positive result, there would need to be around 3000 ppm of Aroclor 1242 in the oil (and again no chance of any other background chlorine). If it were possible to specifically determine PCBs using the Q4000, you would first need to know what Aroclor is present because determination would be based on the percent chlorine on that molecule. For example: Aroclor 1242 is 42% chlorine. 50 ppm PCB of Aroclor 1242 is the equivalent of 21 ppm chloride. But more heavily chlorinated Aroclors would be different.

Once again, I can't stress this enough * you cannot use Clor-D-Tect kits or Clor-N-Oil kits to determine if used oils contain PCBs. There is no field test method to determine PCBs in used oils * lab analysis is the only option.

Please let me know if you have any other questions.

Kind regards,

Cathy Kopylec

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>>> "WALL Dave" <WALL.Dave@deq.state.or.us> 03/17/10 10:39 AM >>>
I would appreciate your help to determine the approximate accuracy of three of your products.

Let's say, for example, you had three known PCB concentrations in used oil:

400 ppm, 800 ppm, and 2000 ppm.

What kind of reading would be expected as a result, when using the CLOR-D-TECT 4000 on the above known quantities?

What about with the CLOR-D-TECT 1000 or the CLOR-N-OIL test kits?

Thank you for your help! I look forward to your quick response.

David Wall

Hazardous Waste Compliance Inspector

The Oregon Department of Environmental Quality

503-229-6385

HydroCLOR-Q (HY-DRO)



Analytes Chlorinated Organics

Matrix Water/Oil Mix, Antifreeze

Detection Method Quantitative colorimetric titration

Action Levels 200-4000 ppm

Analysis Time Less than 10 minutes

Catalog Number HY-DRO

Packaging Sold in packs of 12

Pricing:

Please **log in** or **create an account** in order to purchase products or view pricing.

Field Test kit for Organic Chlorine Determination for Oil/Water Mixtures and Used Antifreeze/Coolant

On-Site Test Kit for Total Organic Chlorine in Waste Water, Used Antifreeze and Bilge water

HydroClor-Q is a quantitative field test kit designed to measure organic chlorine contamination in oil/water mixtures and used antifreeze. The kit can be used on water-soluble cutting fluids, sump and bilge water, antifreeze or any organic matrix containing water. If water concentration in the sample is greater than 70%, the result can be read directly from the titration burette. For water concentrations less than 70%, please download the conversion chart under "Technical Papers" at the bottom of this page.

HydroClor-Q only measures "organic" chlorine, therefore inorganic chloride from seawater or other sources will not interfere with the test.

The pocket-sized kit is easy to use and comes complete with everything necessary to perform one test. All premeasured reagents are sealed in glass ampules. The test takes less than 10 minutes to run and quantifies total organic chlorine in the range of 200 ppm to 4000 ppm. This kit is extremely useful for identifying chlorinated solvent contamination in water and wastewater.

Clor-D-Tect Q4000 (Q4-000-SG)



Pricing:

Please **log in** or **create an account** in order to purchase products or view pricing.

Analytes	Chlorine, Chlorinated Organics	
Matrix	Used Oil, Solvents, Organic Liquids	
Detection Method	Quantitative colorimetric titration	
Action Levels	200 - 4000 ppm	
MDL	200 ppm	
MQL	600 ppm	
Interferences	Sulfur may cause false positive results	
Overall Accuracy	10% +/- MDL	
Analysis Time	5 minutes	
Catalog Number	Q4-000	
Packaging	20 Kits to a shelf pack, 80 kits per case. Minimum order 10 kits. Orders greater than 10 kits must be in multiples of 20.	



Field test kit for quantifying total chlorine (halogens) in used oil

On-Site Test Kit for Quantifying Total Chlorine (halogens) in Used Oil (waste oil)

U.S. EPA SW-846 Method 9077 ASTM Method D-5384

Clor-D-Tect Q4000, under USEPA SW-846 Method 9077, is used to determine quantitative chlorine levels in used oil. The kit determines the chlorine level of used oil over the range of 200 ppm to 4000 ppm. The kit has been proven invaluable when different oil lots are blended or when the user must know how close a quantity of waste oil is to the 1000 ppm or 4000 ppm action level. Clor-D-Tect Q4000 is a proven test to measure chlorine in crankcase, hydraulic, diesel, lubricating oils and virtually any hydrocarbon based solvent.

The pocket-sized Clor-D-Tect Q4000 kit has all its premeasured reagents sealed in glass ampules for safe, consistent, and accurate results. The test is quick and easy to run, with results obtained in the field in less than 5 minutes with no special training.



732 Methods of Testing for Chlorinated Compounds in Used Oil

by Dexsil Corp.

Dexsil Corp. is a manufacturer of test kits for a variety of industrial and environmental analyses. The company is based in Hamden, Conn.

Introduction

In order to reduce the amount of hazardous halogenated compounds in the atmosphere, federal regulations (40 CFR 266 recodified at 40 CFR 279; see 9[510) place strict limits on the amounts of halogenated materials that are allowed in used oil burned for energy recovery. Used oil may contain up to 1,000 parts per million (ppm) total halogens or, in some cases, up to 4,000 ppm if it can be shown that the source of the halogens is non-hazardous.

With millions of gallons of used oil being collected every year for reuse as a fuel, there is a great need for reliable test methods to determine if a batch of oil is in compliance with the regulations. Several methods exist for determining the amounts of halogenated compounds in used oil. Some of the methods can be used on site and some require that a sample be sent to a laboratory. Even though the regulations limit the concentration in used oil of all halogenated compounds, they only require that testing be done for chlorinated compounds, so all of the methods discussed here test for chlorinated compounds only, unless otherwise noted.

The six methods of analysis that will be discussed include:

- microcoulometry;
- x-rav fluorescence:
- gas chromatography:
- · Beilstein test;
- halogen sniffers: and
- · Chemical test kits.

The first three methods use sophisticated instruments and generally require that they be performed in a laboratory. The latter three are field methods that are portable enough to be used on site. The laboratory methods require a greater level of operator expertise, but supply a higher level of precision as well as a lower limit of detection.



MICROCOULOMETRY

Microcoulometers are capable of testing almost any type of material for total chlorine content. A small amount of sample (less than 10 milligrams) is either injected or placed into a quartz combustion tube where the temperature ranges from 600' to 1,000' C. Pure oxygen is passed through the quartz tube and any chlorinecontaining components are combusted completely. The resulting combustion products are swept into a titration cell where the chloride ions are trapped in an electrolyte solution. The electrolyte solution contains silver ions that immediately combine with any chloride ions and drop out of

solution as insoluble silver

chloride. A silver electrode in the titration cell electrically replaces the used up silver ions until the

concentration of silver ions is back to where it was before the titration began. By keeping track of the amount of current needed to generate the required amount of silver, the instrument is capable of determining how much chlorine was present in the original sample. Dividing the total amount of chlorine present by the weight of the sample gives the concentration of chlorine that is actually in the sample.

Microcoulometers can detect chlorine (and bromine) concentrations in used oils from the low ppm range up to percentages if dilutions are performed. A trained analyst is required to operate the instrument and calibrations must be performed regularly. After the microcoulometer is turned on, it takes about an hour to warm up before the first analyses can be run. Microcoulometers cost about \$15,000. Microcoulometry has received an EPA method designation of 9076 in the SW-846 manual.

X-ray Fluorescence

X-rav fluorescence (XRF) instruments also are used primarily in the laboratory although some are portable. The sample to be analyzed is placed in a sample cup which has a transparent bottom. The sample cup is then placed in the analyzer where it is irradiated with X-rays of a specific wavelength. Any chlorine atoms in the sample absorb a portion of the X-rays and then emit radiation back out of the sample cup at a wavelength specific for chlorine. A detector in the instrument then quantifies the amount of radiation coming back from the chlorine atoms and. by knowing the surface area that is exposed, can then calculate the concentration of chlorine in the sample.

The technique requires a trained analyst and is subject to some interference from other elements such as sulfur. A sample which is not homogeneous and is subject to settling in lavers may give incorrect results because the instrument only reads the bottom layer of the sample. XRF analyzers range from about \$30,000 for single. element analyzers to hundreds of thousands for sophisticated multi-element instruments. XRF analysis of waste oil has been assigned EPA SW-846 method number 9075.



Gas Chromatography

Gas chromatography (GC) is a laboratory method, and is the only method discussed here that can actually quantify specific halogenated compounds. It is not generally used to test used oils for total halogens because it cannot detect inorganic halogen contamination. GC is often misinterpreted as being an exhaustive test for halogen contamination but cannot be used for classifying whether used oils contain less than or more than 1,000 ppm total halogens. It is the only one of these methods that can be used, however, when looking for a specific halogenated organic compound.

Beilstein Test

The Beilstein, or copper wire, test is the simplest of the methods discussed here. When a compound containing chlorine is burned on a copper surface it emits a green flame that easily can be detected by eve when the chlorine is in sufficient concentration. Copper wire or copper '@wool" provides enough surface area to perform the test. The Beilstein test only (,lives a qualitative reading and is only useful in the percentage range, not at ppm levels. It will not detect chlorine as low as the regulated limits, but will give an indication of gross contamination. Because the sample must be ignited, the test never should be- run in the vicinity of flammable materials. This method is very inexpensive, but it is not quantitative, is not approved, and is dangerous. It should only be used when other methods are not available.

Halogen Sniffers

The small, hand-held devices most often referred to as "sniffers" have some applications with used oils, but the user must be careful not to read more into the result than what is actually found. These instruments use a variety of detection techniques, but all of them rely on detecting the presence of halogenated compounds in the headspace above the used oil being analyzed. They easily find volatile compounds that are not halogenated. The instruments are not quantitative because they can only measure what is in the air space above the oil, not what is in the oil itself. The sniffers are inexpensive, costing a few thousand dollars, and are easily portable. Because they are not specific and not quantitative, the sniffers are not EPA-approved, and therefore should not be used to verify compliance with the regulations.

Chemical Test Kits

The portable chemical test kits are designed to test for all sources of chlorine in a sample of used oil, and are easy to use. A sample of oil is taken with a polypropylene syringe and introduced into a plastic test tube. Glass ampules in the test tube contain a diluent and a small amount of metallic sodium. The sodium reacts with any compounds that contain halogens and removes the halogens from their organic backbones. The resulting chloride ions are then extracted into an aqueous buffer solution where they can be easily measured by the use of a calorimetric indicating



reagent. Kits are available to provide either a "go, no-go" answer at the 1,000 ppm level or to give a quantitative result over the range of 200 to 4,000 ppm.

The kits are inexpensive, at about \$5.00 per test, and are designed to be run by anyone. However, as with any analytical test care must be taken to ensure that the test kits are used correctly. The test kits have recently been assigned EPA SW-846 Method number 9077. The kits are applicable for most types of used oil, however oils that contain more than three or four percent sulfur may give false positive (high bias) results.

CONCLUSION

All of the methods discussed above have applications where they are most appropriate. When choosing a method for everyday routine analysis of used oil, make sure to weight all the important variables such as cost, time required, training necessary, reliability, and whether or not the technique has been recognized by EPA. If there is ever a discrepancy between test methods, it is always to the tester's advantage to have used a test method, which is recognized by EPA. Keep in mind that EPA methods do not in themselves guarantee good data--the tests must be run properly, samples must be taken and stored correctly, and results must be recorded accurately. If the proper time and care is invested early in developing a complete testing policy, the testing procedures will pay for themselves many times over.

SUGGESTIONS FOR USING THE HYDROCLOR-Q TEST KIT

HydroClor-Q is designed for use on water and oil/water mixtures. For accuracy, the sample should contain more than 70% water. Samples containing more oil (less water) may be tested accurately if the following correction is used.

For example, if the sample contained 6 ml water and 4 ml oil (60% water) and the reading off the syringe was 2000 ppm, then the true concentration would be:

2000
$$\underline{10 + 4}_{\bullet} = 2800 \text{ ppm}$$

For samples that contain >80% oil, use Clor-D-Tect Q4000, designed for used oil analysis.

CONVERSION FACTORS FOR HYDROCLOR-Q

Divide result obtained by appropriate conversion factor

Percent Water	Percent Oil	Conversion Factor
100	0	1
95	5	0.975
90	10	0.95
85	15	0.925
80	20	0.9
75	25	0.875
70	30	0.85
65	35	0.825
60	40	0.8
55	45	0.775
50	50	0.75
45	55	0.725
40	60	0.7
35	65	0.675
30	70	0.65
25	75	0.625
20	80	0.6
15	85	0.575
10	90	0.55
5	95	0.525
0	100	0.5